

Hydration Dynamics Observed by *In-situ* X-ray Diffraction from Ni-Fluorohectorite

E. DiMasi (BNL), J. O. Fossum, T. N. Aalerud, S. B. Lutnaes (Norwegian Univ. of Sci. and Techn. (NTNU), Norway), G. J. da Silva (NTNU and Universidade de Brasília, Brazil), K. J. Maloy (Univ. of Oslo, Norway), K. Knudsen (Institute for Energy Technology (IFE), Norway)
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Introduction: Clay minerals are unique among lamellar solids in their capacity to be pillared by a wide variety of intercalated cations. This allows the sorption capability of the clay to be tuned, in turn. The hydration dynamics of swelling clays is of particular interest, since the intercalated cation affects both the time dependence of water absorption and the structural changes that accompany hydration.

Methods and Materials: We have performed *in-situ* x-ray diffraction measurements of synthetic fluorohectorites having either Na^+ or Ni^{2+} as the intercalated cation. The samples are pressed powders, aligned along the layer stacking direction. Changes in the basal (00L-type) Bragg peak positions occur as layers of water molecules enter between the silicate sheets. The hydration proceeds as the temperature is changed under conditions of controlled humidity [1,2].

Results: Figure 1(a) shows diffraction from the (001) peak for Ni-fluorohectorite under humid conditions. Curves from blue, through green to red and finally black show the shift of (001) intensity to higher q , as water leaves the clay and the interlayer spacing decreases. The Ni sample shows well resolved (001) peaks in its dry state and its fully hydrated state, which contains about 3 layers of intercalated water. However, the intermediate hydration states are not well separated from each other, and show considerable intensity between the Bragg peak positions. This shows that disordered intercalation occurs, with alternate stacking of layers containing different amounts of water [3]. For the Na fluorohectorite, the results differ considerably in that only three hydration states are observed, with only 2 water layers in the fully hydrated state. Signatures of mixed intercalation occur only for the transition from 1-2 water layers, and the peak shifting is considerably less than for the Ni case.

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References:

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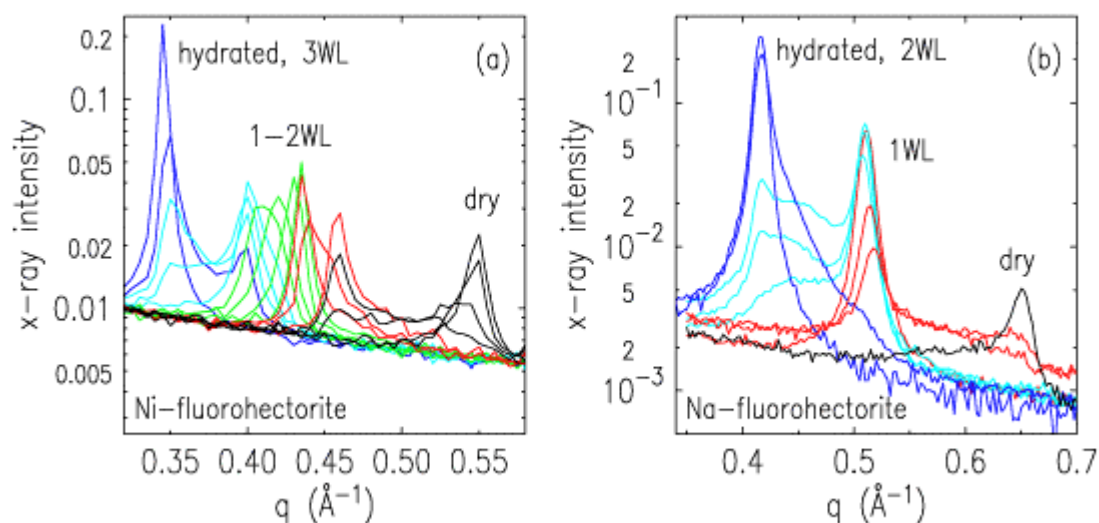


Figure 1. (a) (001) peak intensity from Ni-fluorohectorite under humid conditions as temperature is raised from 10°C to 130°C (lowest temperatures are blue and proceed through green, red and black). (b) The simpler case of hydration in Na-fluorohectorite, with only three hydration states and a maximum hydration of two water layers.